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Energy Storage in Condensed Media via Charge Separation and Trapping

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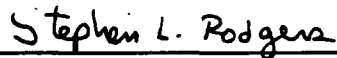Edwards AFB CA 93523-5000

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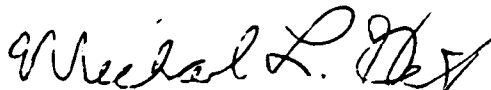
FOREWORD

This final report was submitted by the Department of Chemistry, University of California, Irvine CA on completion of contract F04611-87-K-0024 with the Astronautics Laboratory (AFSC), Edwards AFB CA. AL Project Manager is Lt Roeland van Opijnen.

This report has been reviewed and is approved for release and distribution in accordance with the distribution statement on the cover and on the DD Form 1473.

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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Principles of charge separation, and storage in solids have been developed. The storage density of ion hole pairs is inversely proportional to storage time. Number densities as high as $10^{18} cc^{-1}$ can be stabilized in xenon, at higher densities radiative ion-hole pair recombination leads to loss through laser action. The first solid state exciplex lasers have been demonstrated. Experiments and theoretical simulations indicate that atomic radicals can be stored in solids with near theoretical limits. Pressure stabilizes such metastable solids. Atomic Cl/Xe has been demonstrated to be stable under pressure for several months.

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INTRODUCTION

While at the onset, our program was more narrowly defined along the lines of energy storage in solids via the generation and storage of charges, over the course of studies the scope was expanded to address the more general prospects of energy storage in solids. Thus, we have addressed not only the issues of charge storage, but also issues related to the generation and storage of radicals in condensed media. The work was planned to provide a deep understanding of principles by scrutinizing, both experimentally and theoretically, what may be referred to as model systems. I believe that we have achieved these aims in a most satisfactory manner. As is usual in successful scientific research, our work has provided answers to many fundamental questions and raised new ones of relevance to the HEDM program. The main body of this work has already been published. More than twenty-four papers have appeared in scientific journals, and more than five have appeared as conference proceedings. These are listed as references 1-29. In addition to the scientific accomplishments, it is important to note that a significant number of graduate students and postdoctoral fellows have been trained in studies of condensed matter dynamics. Some of these coworkers, upon graduation, have continued to contribute with their own researches to the general mission of HEDM. A list of coworkers, who have at least in part been supported through this contract, is presented in Table I.

Given the volume of accomplishments, I will only highlight our main findings, concentrating mainly on results that relate most directly to practical considerations of the HEDM mission. The storage of energy at high densities is not only relevant to propulsion but also to other applications. When the stored energy is released in the form of photons, as opposed to kinetic energy, the system is then relevant to laser applications. This indeed has been demonstrated in our group, and I will shortly comment on such developments. When the stored energy can be accessed reversibly, then a high density storage system is relevant to optical memory applications. Some of the studied systems can be regarded as models of such applications as well. Finally, in the course of our studies we have developed new concepts, and methodologies for studies of condensed phase dynamics in general, and given a firm footing for the field of reactive molecular dynamics in solids. These developments will have a significant impact on future studies of systems relevant to the HEDM mission in general. In what follows, I will comment on each of these achievements succinctly, for the details of the results reference is made to the published work [1-29].
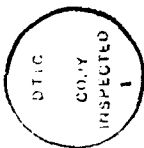
TABLE I

SUPPORTED PERSONNEL

A) Graduate Students

M.E. Fajardo	Ph.D.(1988)
R. Alimi	Ph.D. (1990)
F. Okada	M.S.(1989)
J. Feld	M.S. (1990)
W. Lawrence	Ph.D. candidate
H. Kunttu	Ph.D. candidate
G. Valero	Ph.D. candidate
P. Seed	summer student (1987)

B) Postdoctorals

A. Hayrapetian	6/87 - 9/87
R. Withnall	10/87 - 7/88
M. Fajardo	6/88 - 9/88
A. Becker	3/88 - 3/89
A.I. Katz	4/88 - present
E. Sekreta	3/90 - present

A. Storage of Charge

The most obvious method of charge storage in solids, is by its in-situ generation. The most convenient method for accomplishing this would be by optical means. We have therefore considered in detail the principles of photo-induced charge transfer processes, and the subsequent separation and storage of charges by trapping in the solid. The requirements for effecting this process are a) the accessibility of delocalized charge transfer states, b) a mechanism for trapping of photogenerated charges, c) stable trapping of charges by localization at deep traps. Such a scheme can therefore be accomplished in solids in which both delocalized and localized excitonic states coexist. Secondly, it is necessary for excitonic states to be strongly coupled to the lattice, and finally, the localized charge storage states should possess a large barrier toward recombination. The last two criteria can be met in dielectrics of large bandgap and significant compressibility. The coexistence of both delocalized and localized charge transfer states is a more stringent requirement which is dictated by the electronic structure of the medium, and in particular with the exact description of the valence band. For a most complete discussion of these considerations, see the HEDM conference proceedings [26].

i. Delocalization: We have shown quite generally that, for solids with large valence bands, $B \gtrsim 0.75eV$, delocalized ion-hole states can be accessed optically. Due to the spherically symmetric, Coulombic potential between ion and hole, the stationary states of such a charge pair is described as a Rydberg progression. The principles, the theoretical foundations and experimental verification of this novel concept has been detailed in a series of papers dealing with halogen atom doped solid xenon [3,7,8,10,13,15,20,26]. Since our original work, several others have shown the generality of the concept by observing Rydberg hole progressions in H, OH and likely SF_5, doped xenon [30,31]. While we had predicted that the same consideration should be valid for Kr as well [13], our own experiments on F doped Kr showed that the delocalization is limited to immediate nearest neighbors [20]. However, fluorine is a special case. While F atoms have large electron affinities, due to electron-electron repulsion, F shows a saturation effect: F^- is most stable for the transfer of $0.6e^-$, above which it is destabilized [32]. As a result, even in Xe, the Rydberg hole progression of F doped solids cannot be observed [7]. Using synchrotron radiation, most recently, the Rydberg progression of Cl doped solid Kr has been observed [30] giving full credibility to our previous predictions. No such state is expected in solid Argon, and none has been found. Despite the presence of delocalized ion-hole states in solid Kr, we have not yet been able to effect charge separation in Kr [20]. Evidently, the Rydberg orbits, even near the ionization limit in Kr, are too small for the hole to escape the

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Coulombic field of the negative ion. Thus, it would seem that an effective Onsager escape radius, needs to be reached for charge separation to occur. For ion-hole states to separate, a valence bandwidth of $\geq 1eV$ is discovered experimentally to be the requisite limit. This defines an escape radius of ~10Å.

Beside the rare gases, dielectric solids which fit the above requirements can be found in polyatomics. Both solid hydrogen and nitrogen are such examples. In fluorine doped solid nitrogen, we have observed visible emissions, which we at present attribute to charge transfer states. Our initial investigations have been complicated by impurity emissions. However, delocalization seems to take place; since emission can be induced in a very broad range of excitations within the bandgap. Studies in solid nitrogen, and other molecular solids, such as NO and NH_3, will be useful to make extrapolations to molecular solids in general, and H_2 in particular. Such studies continue in our laboratories.

ii. Charge Separation: The principles of charge separation via self-trapping have been most clearly presented in reference 8. Efficient coupling of delocalized hole states to phonons is required, and a large lattice deformation should accompany the self-trapping process. The lattice relaxation, produces a barrier, E_H, for ion-hole recombination. Thus, although the charges are separated within their mutual Coulombic field, at temperatures where $kT \ll E_H$, no direct recombination can occur. Phonon assisted charge migration remains possible: as the temperature of the solid is increased, the stored localized holes can be set into motion. The result of thermal agitation is radiative ion-hole recombination. We have clearly shown that this process is dominated by either pure tunneling, which gives rise to visible phosphorescence, or phonon assisted tunneling, which can be verified by kinetic analysis of thermoluminescence curves. These latter two mechanisms determine the maximum charge density storable for a given time in the medium (see below).

The depth of the trap for self-trapped holes by lattice relaxation, is similar for all rare gases. However, the barrier to recombination, E_H, is dictated by the valence bandwidth as well as the lattice relaxation energy. The narrower the valence bandwidth, the higher E_H, therefore the more stable is the trapping. Thus, we had expected that self-trapped states would be even more stable in the lighter rare gases, and in molecular solids. This expectation has not been verifiable, since we have not been able to achieve photo-induced charge separation to any appreciable extent in any medium other than xenon. Although it is difficult to make inferences based on negative results, it is at present believed that our inability to separate charges in Kr is due to the reduced extent of delocalization in the Rydberg series of Cl, Br, I doped Kr. To prove this conjecture, a most direct experiment is planned, namely: broadening of the bands in solid Kr by applying pressure to the solid.

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We have already completed the first set of our studies in diamond anvil cells, at pressures as high as 200,000 atmospheres, as a preliminary to studies in Kr [21]. The experiments in Kr are, however, made difficult by the fact that we have to rely on two-photon access of Rydberg states, since direct access of these states is not possible due to the U.V. cutoff of diamond. This consideration will be relaxed at ultra-high pressures, since the charge transfer states undergo a large red shift as a function of pressure, as we have already demonstrated in our studies in Xe [21].

iii. Limitations on Charge Pair Densities: Perhaps the most important question for practical applications, is the limit on storable charge pair density in dielectric solids. As discussed above, the number density of self-trapped hole-ion pairs is limited by the tunneling length of charges. Tunneling rates have exponential distance dependence. As a result, a careful analysis of geometric factors, yields a hyperbolic time dependence for storage density: $\rho \propto 1/t$, and the proportionality is given by tunneling barrier characteristics. We have shown that charge pair densities of order $10^{18} cm^{-3}$ can be maintained for timescales or order of minutes. This number density can be exceeded for very short time storage. As an example, it is possible to generate densities as high as $10^{20} cm^{-3}$ for time scales $\leq 10^{-8}s$. However, at such number densities, radiation and stimulated emission become significant loss channels. Not all is lost. Such systems can be regarded as ideal lasers as we have argued and already demonstrated.

At the present level of our understanding of charge storage in dielectric solids, it seems difficult to extrapolate our results to the possibility of useful propellants. To increase the stored number densities above $10^{18} cc^{-1}$ for useful lengths of time, it would be essential to consider media with much larger dielectric constants. Molecular solids, possessing permanent dipoles, is a possible means of making such an attempt. Solid NO is a candidate system. However, solid hydrogen seems to be a system comparable to xenon, and charge storage experiments in that medium seem to be in good accord with our predictions [33].

B. Storage of Radicals

In order to reach the requisite radical storage densities of order ~1% in solids, it is clear that clever schemes for generation and storage are essential. Again, the most obvious scheme is in situ photogeneration. This requires a deep understanding of photodissociation mechanisms, and recombination dynamics. We have embarked on this task with both experiment and theory. For the first time there seems to be quantitative models and results, with which the depth of our understanding can be tested. In the published work to date,

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we have considered the dynamics of direct dissociation of HI in Xe [6,11,34], and F_2 in Ar and Kr [19,20,22,24], and the charge transfer induced dissociation of Cl_2 [15,21,10]. The theory on these systems has been carried out in collaboration with R.B. Gerber.

·   i. HI: The photodissociation of HI is a prototype of a system that can produce, and lead to the storage of, H atoms in solids. As such, it is of direct relevance to HEDM considerations. We have shown in theoretical simulations, that at least in heavy hosts, the H atom exits the trap site with near thermal energy, and traps in an O_h site adjacent to the original trap [6]. The trapping site of H atoms, has recently been verified by photodissociation studies of hydrocarbons, NH_3, and H_2O in solid xenon [35]. This would imply that H atom densities as high as 1 per original parent donor can be created in such solids. The results of these simulations are subject to some doubt for mainly two reasons, the validity of pair potentials used, and the fact that the simulations are conducted by classical mechanics (ignoring zero point energy and tunneling dynamics). It has therefore been essential to verify these expectations experimentally. Our initial studies in crystalline Xe at high temperature, showed that indeed the quantum yields of dissociation were a strong function of temperature [11], which is a signature of the fact that the H atom motion is dictated by the lattice and not the kinetic energy of H. More recent measurements in our laboratory have shown the non-monotonic temperature dependence of photodissociation quantum yields, the yields show a minimum near 35K, as predicted by the theory [34]. Thus, we have strong reasons to believe that the main features of the theory are valid. The HI-Xe system is however complicated experimentally, since charge transfer states come to play. In fact we have observed two new charge separated states in these media. In concentrated HI/Xe solids, dimerization is not possible to completely eliminate. In these solids we observe a new thermoluminescence peak which is even brighter than the I^- + hole recombination. We have ascribed this green emission to I_2^- + hole. Spectroscopic validation of this assignment is not yet complete. In dilute HI/Xe solids, we observe two new fluorescence features at 750 and 380nm, respectively. These fluorescence features follow the HI dissociation dynamics exactly and through the kinetic analysis are ascribed to H atoms bound at self-trapped hole sites i.e. H^+ in Xe. These are indeed exciting discoveries since they generalize the charge separation and storage dynamics to diatomic ion cores and impurity stabilized hole traps.

ii. F_2: The overlap between experiment and theory has been most complete for the photodissociation of F_2 in solid Ar and Kr. Almost all of the theoretical predictions have been at least qualitatively verified. These include:

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a) F_2 dissociation quantum yields reach unity for excess energies above ~2eV, and dissociation is possible at all accessible absorption wavelengths [22,24].

b) F atoms upon dissociation at excess energies of $\geq$ 2.4eV undergo long range migration of order 10 lattice sites. At low excess energies, F atoms trap in O_h interstitial sites adjacent to the original trap cage [14,20].

c) Photodissociation quantum yields are higher at 4 K than at 12 K, due to the fact that at the lower temperature F freezes in a librational well prealigned with the reactive cone of the cage [24].

d) F atoms can only trap at interstitial O_h sites. This was shown experimentally by excitation spectral analysis in $F_2/Kr/Ar$ solids [20].

Thus, it would seem that in this case, it is possible to predict the photogeneration dynamics. It is for example possible to predict that for dissociation at excess energies of ~1eV, F atoms can be generated at a density of 1 per O_h site. Hence, number densities of ~10% should be quite feasible. We intend to verify such predictions in an attempt to prepare metastable solids. Clearly, one needs to understand the recombination dynamics of trapped atoms to answer such questions in full. Experiments addressing this issue have already been completed.

Recombination of F atoms in solid Kr were extensively investigated [36]. These studies are achieved by fully dissociating F_2, and monitoring the Xe_2F emission while the sample is heated. The resultant thermograph is then analyzed by kinetic models. It is now clear that neither a first order recombination (rate, limited by detrapping) nor a second order recombination (rate, determined by diffusion) can fully account for the observed curves. A quantitative analysis requires one of two models, autocatalysis (upon recombination of a pair, other F atoms are set into motion) or cooperative motion (only mobile F atoms may recombine when a congruent cavity is encountered). This is interesting for HEDM purposes, since it would imply that at low temperatures such metastable solids can be maintained for long times. However, the implication is also that such systems will be susceptible to thermal runaway.

iii. Cl_2: The photodissociation of Cl_2 in rare gas solids has been the subject of many studies. In general there is agreement that excitation into the first absorption continuum does not lead to any dissociation of the molecule. We had however shown that if a charge transfer state between the molecule and medium is accessed optically, e.g., Cl_2^- - Xe^+, then the molecule falls apart with near unit quantum yield. This, we have verified to be the case not only for Xe but also for Kr [7]. Charge delocalization among the trap atoms holds the key to this cooperative dissociation mechanism as well. A theoretical model for this

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process was presented [15]. Most impressively, we have shown that it is possible to carry out dissociation via these charge transfer states to completion even under pressures in excess of 50,000 atmospheres. This, therefore, is a very important photodissociation mechanism since in very dense media it is operative and the dissociation products do not migrate any further than the immediate second nearest neighbor.

The questions as to the highest achievable number densities, and the storage length of time, have led us to consider the effects of pressure on such compressible solids. We have shown that at pressures in excess of 50,000 atmospheres, such solids can be maintained for lengths of time in excess of ~ 1 month The number density in the case study was dictated by the initial doping level to Cl:Xe, 1:250.

These studies clearly show that under proper conditions, radical densities near the theoretical limit should be possible to generate. Note that for interstitial O_h trapping, as in the case of F, this limit corresponds to 50%, $i.e.$ a number density of ~ 1×10^{22}. Under the proper conditions of temperature and pressure, it is also clear that such solids can be maintained for any desired length of time. Investigations in propulsion candidates, should be undertaken.

C. Solid State Exciplex Lasers

Although not directly addressing the HEDM mission, storage of energy at high densities has obvious implications with respect to laser applications. The difference being that in such cases the stored energy is extracted as photons, as opposed to kinetic energy. Such has been the case for the storage of charge transfer complexes of Rg^+X^- in crystalline rare gas solids [1,2,14,16]. We have already demonstrated several high gain laser systems in the published literature: these include XeF in Ar in which the $D \rightarrow X$ laser at 286 nm is shown to be tunable for ~6 nm [16], the $B \rightarrow X$ at 404nm, and the $C \rightarrow A$ at 540 nm is shown to be tunable for ~ 70 nm [14]. We have since observed superradiance in XeF $(D \rightarrow X)$ in solid Kr at 303 nm [36].

Development of these systems as practical sources is now being pursued by several other laboratories. We will pursue aspects of these studies under funding from NSF.

D. General Impacts

In the three year course of our investigations, we have developed several concepts of fundamental interest and demonstrated the generality of principles by applications to problems of fundamental interest. Among these are:

i) The concept of Rydberg holes was discussed above in some length. This was a novel concept which I suspect will lead to a different outlook on excitonic dynamics in dielectrics (be it solid or liquid).

ii) The condensed phase laser induced harpoon process, is a novel formulation that has led to some of the more important results in solid state energy storage. It also had proven to be a useful tool for investigations of liquid phase and high pressure gas phase studies. These, we have already demonstrated in several publications [5,12,17,18].

iii) Charge transfer states as a tool for detecting halogen atoms, was developed in our solid state studies. However, we have shown its utility in other media. The most important of these applications was recently demonstrated in gas phase studies of OClO dissociation, where we categorically demonstrated the absence of any role due to the photochemistry of OClO in the destruction of stratospheric ozone (the Antarctic ozone hole in particular) [23].

iv) Concepts relating to cooperative photodynamics in condensed media have taken a strong foothold in our studies for the first time.

v) Molecular dynamics studies in crystalline solids, theory and experiment, have been established as an important field. The keys to the HEDM mission, should be sought in the maturation of the field.

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